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REAFFIRMED
2003

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 67]

FOAMING CHARACTERISTICS OF LUBRICATING OILS

(First Revision)

UDC 621·892 : 532·694·1

Adapted from the Joint Publication ASTM Designation D 892-79 and IP Designation 146/80.

(First Reprint AUGUST 2000)

1. SCOPE

1.1 This method is intended for the determination of foaming characteristics of lubricating oils at specified temperatures. Means of empirically rating the foaming tendency and stability of foam are also described.

2. OUTLINE OF THE METHOD

2.1 The sample, maintained at a temperature of 24°C, is blown with air at a constant rate for 5 minutes and then allowed to settle for 10 minutes. The volume of foam is measured at the end of both periods. The test is repeated on a second sample at 93°C and then, after collapsing the foam, at 24°C.

3. APPARATUS

3.1 The following apparatus is required.

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Gr 4 *July* 1982

3.1.1 *Foaming Test Apparatus* — as shown in Fig. 1, consisting of a 1 000-ml measuring cylinder and an air-inlet tube to the bottom of which is fastened a 25·4 mm diameter spherical gas diffuser stone*, as prescribed in **3.1.1.1** and made of fused crystalline alumina grain. The diameter of the cylinder shall be such that the distance from the inside bottom to the 1 000-ml graduation mark is 360 ± 25 mm. It shall be circular at the top (*see* Note) and shall be fitted with a rubber stopper having one hole at the centre for the air-inlet tube and a second hole off-centre for an air-outlet tube. The air-inlet tube shall be so adjusted that when the rubber stopper is fitted tightly into the cylinder, the diffuser stone just touches the bottom of the cylinder and is approximately at the centre of the circular cross-section. Diffuser stones may be attached to air-inlet tubes by any suitable means. A typical arrangement is shown in Fig. 2.

NOTE — Graduated cylinders with circular tops may be prepared from cylinders with pouring spouts by cutting them off below the spouts.

3.1.1.1 The diffuser stone shall meet the following requirements when tested in accordance with the method given in Appendix A:

Characteristic	*Requirement*
Diameter of stone, mm	25·4
Pore diameter, microns, *Max*	80
Permeability at a pressure of 250 mm of water, ml of air per minute	3 000 to 6 000

3.1.2 *Test-Baths* — of suitable types (preferably oil-baths), large enough to permit the immersion of the cylinder at least to the 900-ml mark and capable of being maintained at temperatures constant to ± 0·5°C at 24°C and 93°C respectively. Both the bath and bath liquid shall be clear enough to permit observation of the graduations on the cylinder.

3.1.2.1 Heat-resistant cylindrical glass jars, approximately 30 cm in diameter and 45 cm in height, make satisfactory baths.

3.1.3 *Air-Supply* — shall be from a source capable of maintaining an air flow rate of 94 ± 5 ml per min through the gas diffuser stone. The air shall be passed through a drying tower 300 mm in height packed as follows :

Just above the constriction place a 20 mm layer of cotton wool, then a 110 mm layer of desiccant, a 40 mm layer of indicating desiccant,

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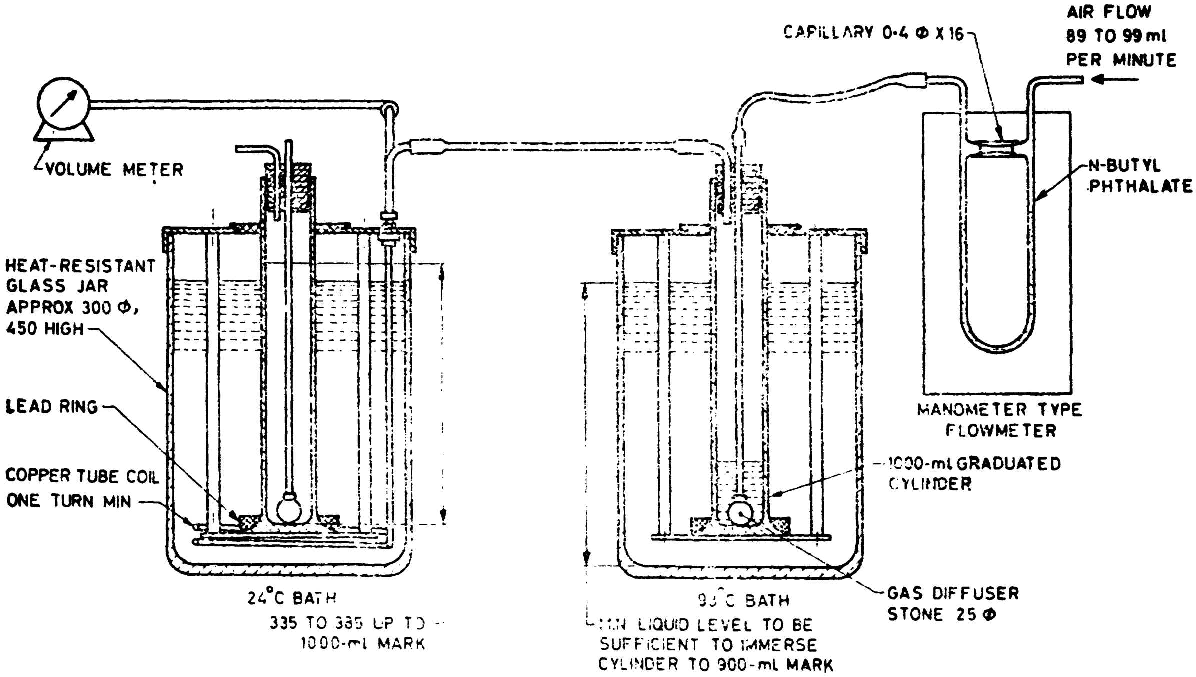
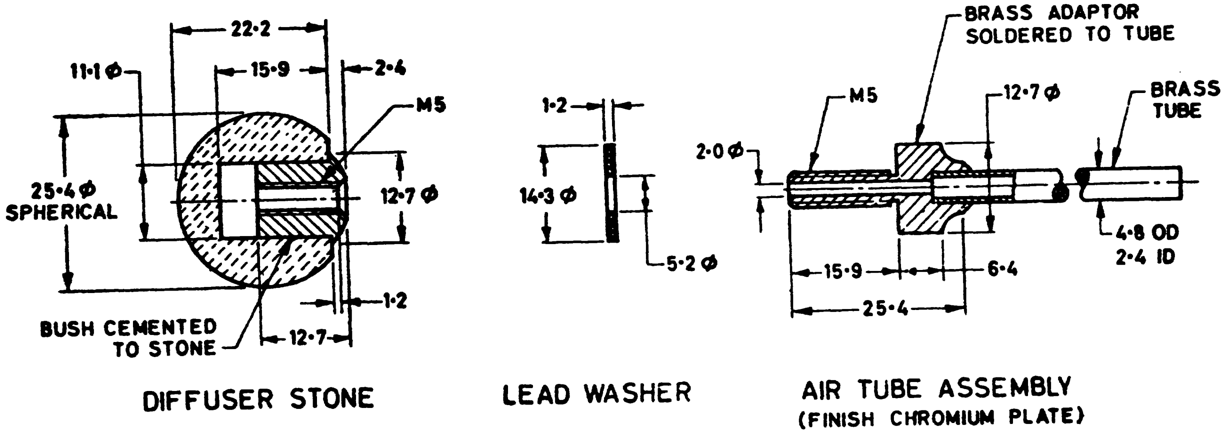
*Suitable diffusers may be obtained from the Norton Co Refractories Division Worcester. Mass 01606 USA. No. MF-45239, P 2180 grade, Alundum porous spheres.


AIR FLOW
89 TO 99 ml
PER MINUTE
CAPILLARY 0·4 Φ X 16
N-BUTYL PHTHALATE
MANOMETER TYPE FLOWMETER
VOLUME METER
HEAT-RESISTANT GLASS JAR APPROX 300 Φ, 450 HIGH
LEAD RING
COPPER TUBE COIL ONE TURN MIN
24°C BATH
335 TO 385 UP TO 1000-ml MARK
93°C BATH
MIN LIQUID LEVEL TO BE SUFFICIENT TO IMMERSE CYLINDER TO 900-ml MARK
1000-ml GRADUATED CYLINDER
GAS DIFFUSER STONE 25 Φ

All dimensions in millimetres.

FIG. 1 FOAMING TESTS APPARATUS



DIFFUSER STONE

LEAD WASHER

AIR TUBE ASSEMBLY
(FINISH CHROMIUM PLATE)

All dimensions in millimetres.

Fig. 2 A Typical Arrangement of Diffuser Stone for Air-Inlet Tubes

a 30 mm layer of desiccant and a 20 mm layer of cotton wool, the cotton wool serves to hold the desiccant in place. Refill the tower when the indicating desiccant begins to show presence of moisture. Any type of flowmeter sensitive to the required tolerance may be used to measure the air flow (*see* Note).

NOTE — A manometer type flowmeter, in which the capillary between the two arms of the U-tube is approximately 0·4 mm in diameter and 16 mm in length, and in which *n*-butylphthalate is the manometric liquid, is suitable.

3.1.3.1 The total volume of air leaving the foaming test apparatus shall be measured by a volume measuring device (*see* Note) capable of accurately measuring gas volumes of about 470 ml. The air shall be passed through at least one loop of copper tubing placed around the inside circumference of the cold bath so that the volume measurement is made at approximately 24°C. Precautions shall be taken to avoid leaks at any one point in the system.

NOTE — A wet test meter calibrated in hundredths of a litre is suitable.

3.1.4 *Timer* — any timing device graduated and accurate to one second or better may be used.

3.1.5 *Thermometer** — One thermometer complying with the following requirements is preferable. If such a thermometer is not readily available, any other thermometer suitable for measuring the temperature of the oil sample and the bath at 24°C and 93°C with an accuracy of ± 0·2°C may be used:

Characteristic	*Requirement*
Range	–20°C to 102°C
Graduation	0·2°C
Immersion, mm	Total
Overall length	415 to 425 mm
Stem diameter	6 to 8 mm
Bulb shape	Cylindrical
Bulb length	15 to 20 mm
Bulb diameter	6 mm but not greater than stem
Length of graduated portion	305 to 350 mm
Distance, bottom of bulb to – 20°C mark	35 to 50 mm
Longer lines chamber	1°C
Figured at each	2°C
Expansion at each	To allow heating to 150°C
Top finish	Ring
Scale error not to exceed	± 0·1°C

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*Thermometer with the Institute of Petroleum Designation IP 64 C conforms to these requirements.

4. MATERIALS

4.0 Caution — For safety precautions, while handling the following reagents, *see* Appendix B.

4.1 Petroleum Distillate — conforming to the following requirements :

Characteristic	*Requirement*	*Method of Test, Ref to IS : 1448**
i) Distillation characteristics:		P : 18
a) 50 percent recovery temperature, °C, *Min*	176	
b) 90 percent recovery temperature, °C, *Min*	190	
c) Final boiling point, °C, *Max*	210	
d) Residue, percent by volume, *Max*	1·5	
ii) Flash point, °C, *Min*	38	P : 20
iii) Copper strip corrosion at 100°C	Not more than slight tarnish	P : 15
iv) Doctor test	Negative	P : 19
v) Acidity of the residue after distillation	No acid reaction to methyl orange	—
vi) Colour, Saybolt	Not darker than †21	P : 14
vii) Sulphuric acid absorption percent by volume, *Max*	5	(*See* Note)

NOTE — Under preparation. Till such time ASTM D 484-76 may be followed.

4.2 Toluene — nitration grade or equivalent (*see* IS : 537-1967†)

4.3 Acetone — (*see* IS : 170-1976‡).

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*Methods of test for Petroleum and its products.

†Specification for toluene, pure nitration grade (*first revision*).

‡Specification for acetone (*second revision*).

4.4 Petroleum Hydrocarbon Solvent (60/80) — conforming to the following requirements:

Characteristic	*Requirement*	*Method of Test, Ref to IS : 1448**
i) Specific gravity at 15·5/15·5°C	0·680 to 0·690	P : 32
ii) Distillation range:		P : 18
a) Initial boiling point, °C, *Min*	55	
b) 90 percent recovery temperature, °C	60 to 80	
c) Final boiling point, °C, *Max*	90	
iii) Existent gum, mg/100 ml of sample, *Max*	2	P : 29
iv) Bromine number, *Max*	1	P : 44
v) Doctor test	Negative	P : 19
vi) Colour	Water, white	P : 13

5. PREPARATION OF APPARATUS

5.0 Thoroughly clean the test cylinder and air-inlet tube after each test to remove any additive(s) remaining from previous tests which would seriously interfere with the results of subsequent tests.

5.1 Cylinder — Rinse the cylinder in turn with chromic acid, petroleum hydrocarbon solvent, distilled water and acetone and then dry by blowing clean and dry air through it.

5.2 Diffuser Stone — Clean the diffuser stone by washing in turn with petroleum distillate, toluene and petroleum hydrocarbon solvent. Immerse the stone in about 300 ml of petroleum distillate and flush a portion of it back and forth through the stone at least five times with vacuum and air pressure. After completing the final washing in petroleum hydrocarbon solvent, dry both tube and stone thoroughly by blowing clean air through them. Wipe the outside of the air-inlet tube, first with a clean cloth moistened with petroleum distillate and then with a clean dry cloth.

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*Methods of test for petroleum and its products.

6. PROCEDURE

6.1 Sequence I — Without mechanical shaking or stirring, decant approximately 400 ml of sample into a beaker. Heat to 49 ± 3°C and allow to cool to 24 ± 3°C. See Option A for stored sample.

> NOTE — Each step of the procedure described in **6.2** and **6.4** respectively, shall be carried out within 3 h after completion of the previous step. In **6.3** the test should be carried out as soon as compatible with the temperature specification and not more than 3 h after immersion of the cyiinder in the 93°C bath.

6.2 Pour the sample into the 1 000-ml cylinder until the liquid level is at the 190 ml mark. Immerse the cylinder at least to the 900 ml mark in the bath maintained at 24 ± 0·5°C. When the oil has reached the bath temperature, insert the diffuser stone and the air-inlet tube with the air source disconnected, and permit the stone to soak for about 5 minutes. Connect the air-outlet tube to the air volume measuring device. At the end of 5 minutes connect to the air source, adjust the air flow rate to 94 ±5 ml per minute, and force clean dry air through the stone for 5 minutes ± 3 seconds, timed from the first appearance of air bubbles rising from the stone. At the end of this period, shut off the air by disconnecting the hose from the flowmeter and immediately record the volume of foam that is, the volume between the oil level and the top of the foam. The total air volume which has passed through the system shall be 470 ± 25 ml. Allow the cylinder to stand for 10 minutes ± 10 seconds and again record the volume of foam.

6.3 Sequence II — Pour a second portion of sample into a cleaned 1 000 ml cylinder until the liquid level is at the 180 ml mark. Immerse the cylinder at least to the 900 ml mark in the bath maintained at 93 ± 0·5°C. When the oil has reached a temperature of 93 ± 1°C, insert a clean diffuser stone and air-inlet tube and proceed as described in **6.2**, recording the volume of foam at the end of the blowing and settling periods.

6.4 Sequence III — Collapse any foam remaining after the test at 93°C (*see* **6.3**), by stirring, cool the sample to a temperature below 43·5°C by allowing the test cylinder to stand in air at room temperature, then place the cylinder in the bath maintained at 24 ± 0·5°C. After the oil has reached bath temperature, insert a cleaned air-inlet tube and diffuser stone and proceed as described in **6.2**, recording the foam value at the end of the blowing the settling periods.

6.5 Some lubricants with modern additives can pass their foam requirements when blended (with the antifoam properly dispersed in small particle sizes) but fail to meet the same requirements after two or more weeks' storage. (It appears that the polar dispersant additives have the potency to attract and hold antifoam particles, such that the apparent increased anti-foam size results in decreased effectiveness to control foam. However, if the same stored oil is merely decanted and poured into engines, transmissions, or gear boxes and those units operated for a few minutes, the oil again meets its foam targets. Similarly, decanting the stored oil into a blender, followed by agitation as described for *option A* below, redisperses the antifoam held in suspension and the oil again will give good foam control. For such oils, *option A* may be used. On the other hand, if the antifoam is not dispersed into sufficiently small particles when the oil is blended, the oil may not meet its foam requirements. If this freshly blended oil were vigorously stirred according to Option A, it is very possible that the oil would then meet its foam targets whereas the plant blend would never do so. Therefore, it is inappropriate and misleading to apply option A for quality control of freshly made blends.

6.5.1 *Option A* — Clean the container of a 1 000-ml high speed blender* using the procedure given in **5.1**. Place 500-ml of sample measured from 18 to 32°C into the container, cover, and stir at maximum speed for 1 minute. Because it is normal for considerable air to be entertained during the agitation, allow to stand until entrained bubbles have dispersed and the temperature of the oil has reached 24 ± 3°C within 3 h following the agitation (*see* Note), start with testing as specified in **6.2**.

NOTE — In case of viscous oils, 3 h may not be enough to disperse the entrained air. If a longer time is required, record the time as a note on the results.

7. REPORTING

7.1 Report the data in the following manner:

Test	*Foaming Tendency Foam Volume, ml at End of 5 Minutes Blowing Period*	*Foam Stability Foam Volume, ml at End of 10 Minutes Setting Period*
As received		
Sequence I	—	—
Sequence II	—	—
Sequence III	—	—
After agitation (Option A, **6.5.1**)		
Sequence I	—	—
Sequence II	—	—
Sequence III	—	—

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*Waring Blender or equivalent.

7.2 For the purpose of reporting results, when the bubble layer fails to completely cover the oil surface and a patch or 'eye' of clear fluid is visible, the value shall be reported as 'nil foam'.

8. ALTERNATIVE PROCEDURE

8.1 For routine testing a simplified testing procedure may be used. This procedure differs from the standard method in only one respect. The total air volume used during the 5 minutes blowing period is not measured after the air has passed through the diffuser stone. This eliminates the volume measuring equipment and the air-tight connections necessary to carry the exit air from the graduated cylinder to the volume measuring device, but requires that the flowmeter be correctly calibrated and that the flow rate be carefully controlled.

8.2 Results obtained by this procedure shall be reported at alternative method.

9. PRECISION

9.1 For the standard test (*see* **6.1** to **6.4**), results of duplicate tests shall not differ by more than the following amounts:

Test	*Repeatability*	*Reproducibility*
Foam volum at the end of 5 minutes blowing period	*See* Fig. 3	*See* Fig. 4

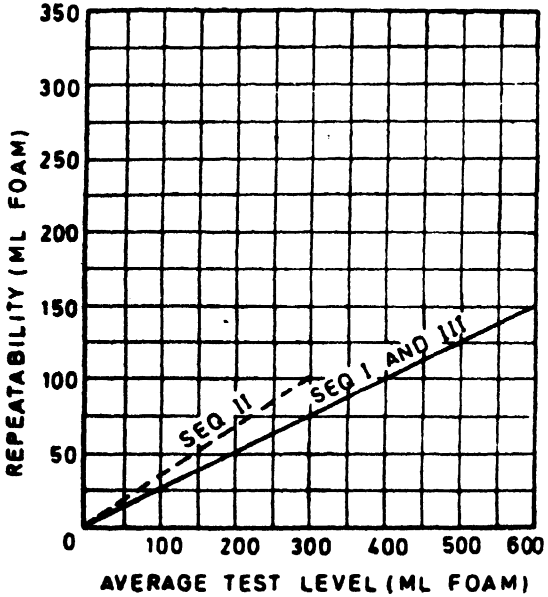

FIG. 3 PRECISION CHART — REPEATABILITY

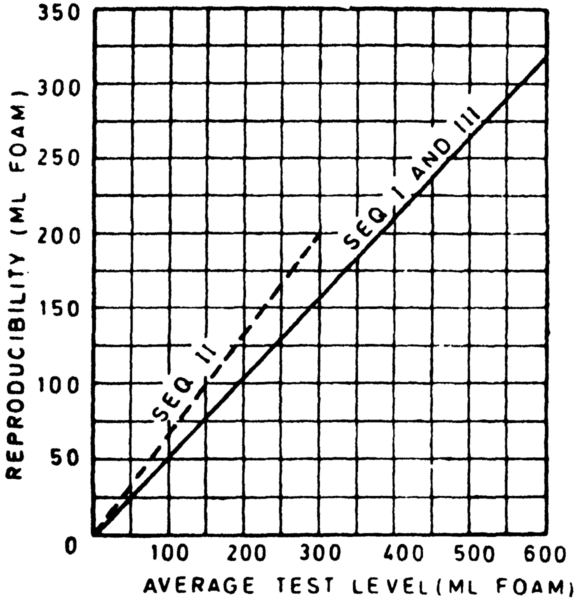

FIG. 4 PRECISION CHART — REPRODUCIBILITY

9.2 For those oils which have been tested by option A (*see* **6.5.1**), no precision data is available.

APPENDIX A

(*Clause* 3.1.1.1)

TEST FOR MAXIMUM PORE DIAMETER AND PERMEABILITY OF STONE DIFFUSERS

A-1. DEFINITIONS

A-1.1 Maximum Pore Diameter — The diameter in micrometers of a capillary of circular cross-section which is equivalent (with respect to surface tension effects) to the larger pore in the diffuser under consideration.

A-1.2 Permeability — The flow of air, in millilitres per minute through the diffuser stone at air pressure of 2·45 kPa (250 mm of water).

A-2. APPARATUS

A-2.1 Apparatus for the maximum pore diameter determination consists of a regulated source of clean, dry, compressed air, a U-tube water manometer of sufficient length to read a pressure differential of 7·85 kPa (800 mm of water) and a cylinder of a size sufficient (250 ml is suitable) to conveniently immerse a diffuser stone to a depth of 100 mm.

A-2.2 Additional apparatus for permeability determination consists of a gas volume meter of sufficient capacity to measure flow rates of at least 6 000 ml/min, and a filtering flask large enough that 25·4-mm diameter diffuser stones will pass through the neck. The flask shall be fitted with a rubber stopper with a single hole to admit the air-inlet tube (*see* Fig. 5).

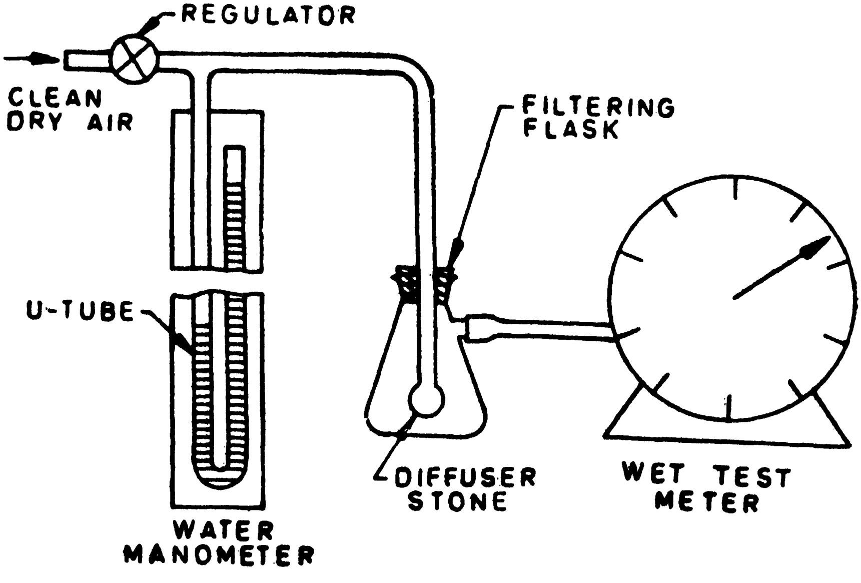

FIG. 5 APPARATUS FOR MEASURING PERMEABILITY

A-3. PROCEDURE

A-3.1 Maximum Pore Diameter — Support the clean diffuser by an air-inlet tube at a depth 100 mm as measured to the top of the stone in distilled water in a cylinder and allow it to soak for at least 2 minutes. Connect the air-inlet tube to a controllable source of clean, compressed air and a manometer as shown in Fig. 6. Increase the air pressure at a rate of about 490 Pa (50 mm of water)/ minute until the first dynamic bubble passes through the filter and rises through the water. The first dynamic bubble is recognized by being followed by a succession of additional bubbles. Read the water level in both legs of the manometer and record the difference *P*. The uniformity of distribution of pores approaching maximum pore size may be observed by gradually increasing the air pressure and noting the uniformity with which streams of bubbles are distributed over the surface. Calculate maximum pore diameter, *D* in micrometers as follows:

$$D = 29\,225\,(P - 100)$$

where P = mm of water.

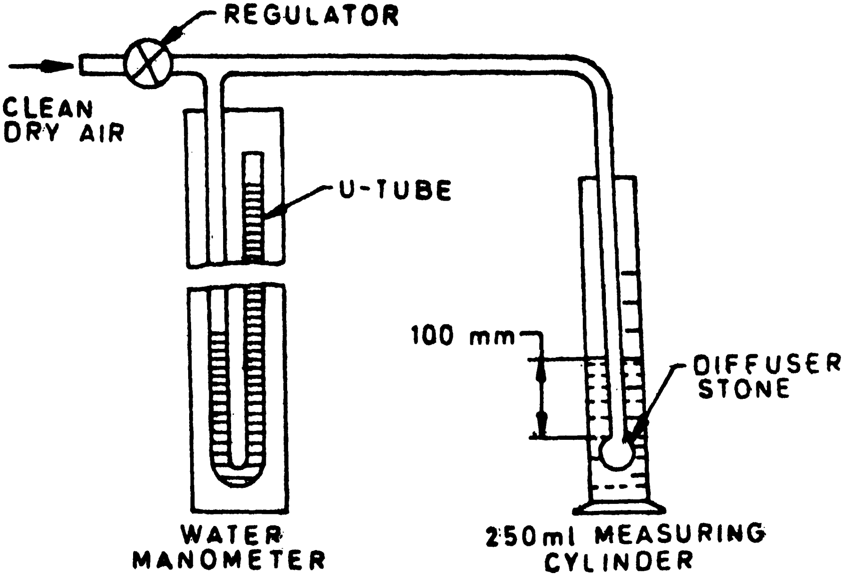

Fig. 6 Apparatus for Measuring Maximum Pore Size

A-3.2 Permeability — Connect the clean, dry diffuser stone with a controllable source of clean, dry, compressed air and place it in a filtering flask connected to a suitable flowmeter as shown in Fig. 5. Adjust the pressure differential to 2·45 kPa (250 mm of water) and measure the rate of flow of air through the diffuser stone in millilitres per minute. Depending on the sensitivity of the flowmeter used, this observation may be made for a suitably longer period and the average flow rate per minute recorded.

APPENDIX B

(*Clause* 4.0)

PRECAUTIONARY STATEMENTS

B-1. PETROLEUM DISTILLATE

Caution : Combustible. Vapour harmful. Keep away from heat and open flame. Keep container closed. Use only with adequate ventilation. Avoid prolonged breathing of vapour or spray mist. Avoid prolonged or repeated contact with skin.

B-2. TOLUENE

Warning — Flammable. Vapour harmful. Keep away from heat, sparks and open flame. Keep container closed. Use with adequate ventilation. Avoid breathing of vapour or spray mist. Avoid prolonged or repeated contact with skin.

B-3. ACETONE

Danger — Extremely flammable. Vapours may cause flash fire. Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Vapours may spread long distances and ignite explosively. Avoid build up of vapours and eliminate all sources of ignition, especially, non-explosion proof electrical apparatus and heaters. Avoid prolonged breathing of vapour or spray mist. Avoid contact with eyes and skin.

B-4. PETROLEUM SPIRIT

Danger — Extremely flammable. Harmful if inhaled. Vapours may cause flash fire. Keep away from heat sparks, and open flame. Keep container closed. Use with adequate ventilation. Avoid buid up of vapours and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters. Avoid prolonged or repeated skin contact.

Reprography Unit, BIS, New Delhi, India

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TO
IS 1448 [P : 67] : 1982 METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P:67]

FOAMING CHARACTERISTICS OF LUBRICATING OILS

(First Revision)

(*Page* 1, *clause* 2.1, *line* 4) — Substitute '93.5°C' *for* '93°C'

(*Page* 1, *clause* 2.1) — Insert new clause after 2.1:

'2.2 The tendency of oils to foam can be a serious problem in system such as high speed gearing, high volume pumping and splash lubricants, inadequate lubricants, cavitation, and overflow failure. This method is suitable in the selection of oils suitable for such operating conditions.'

(*Page* 3, *Fig*. 1) — Substitute 'GAS DIFFUSER STONE 25.4 ϕ' *for* 'GAS DIFFUSER STONE 25 ϕ '.

(*Page* 8, *clause* 6.1, *Note*, *line* 4) — Substitute '93.5°C' *for* '93°C'.

(PCD 1)

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